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Greenwell

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IN THE
PRESENCE OF SUCROSE

BY

EARL EUGENE GREENWELL

THESIS
(Incomplete)

FOR THE

DEGREE OF BACHELOR OF SCIENCE

IN

CHEMISTRY

COLLEGE OF LIBERAL ARTS AND SCIENCES

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THE DETERMINATION OF INVERT SUGAR IN THE PRESENCE OF SUCROSE

I

Introduction

From the work~~s~~ of Soxhlet, Allihn, Browne, and others it has been shown that the amount of reduction in an alkaline tartrate solution of copper sulphate is dependent upon the amounts of invert sugar, sucrose and unreduced copper left in the solution. In the work of Munson and Walker on mixtures of invert sugar and sucrose, two mixtures were used. One mixture contained a total of 0.400 gram of invert sugar and sucrose and the other 2.000 grams of total sugar. With these two mixtures they ran determinations at points 20 milligrams apart, beginning with 20 milligrams and ending with 490 milligrams. Results of all determinations of four series were made into a table.[1] From the averages for each series, a curve was drawn upon cross section paper and by use of these curves the individual determinations that were sufficiently far from the curve to be considered among the less accurate of the determinations made, were eliminated from the final average.

From their remaining results they constructed tables[2] in which the Cuprous-oxide precipitates correspond to certain values of invert sugar in the two mixtures, namely the 0.400 gram and 2.000 grams of total sugar. By observing this table it is seen that the presence of sucrose materially increases the reduction of Fehling's Solution - the amount of increase depending on the ratio of sucrose to invert sugar.

If Browne's formula[3], ($\frac{\text{milligrams sucrose}}{\text{milligrams glucose} + 40} = \text{Const.}$) holds for invert sugar as well as for glucose, and ~~which~~ we assume it does, it may be shown mathematically that determinations of the reducing power of invert sugar in the presence of three different proportions of sucrose will enable us to calculate its reducing power in the presence of all other proportions of sucrose. Since the work of Munson and Walker is tabulated for two values of total sugar (and hence two values of sucrose for each value of invert sugar) it should be necessary to add but one more column to their table to make the method perfectly general. It is therefore proposed in this paper to run a series of determinations of the reducing power of invert sugar in the presence of one gram of total sugar, then extend the results by suitable methods of calculation to secure a perfectly general formula.

II

Discussion of Experimental Details

Before proceeding with the actual carrying out of this work, preliminary calibrations, standardizations and preparation of reagents etc. were necessary. In this connection asbestos ~~sou~~p was prepared by the method required by Munson and Walker. Fehling's Solution was made up according to specifications. Pure sucrose was prepared by three recrystallizations of cane sugar with absolute ethyl-alcohol from the hot saturated aqueous solution of sugar. From this purified sucrose the invert sugar used in the determinations was prepared by either the hot or cold inversion. The inversion by the hot method was found to give the more concordant results. This method consisted in weighing out the amount of sucrose calculated to give the desired amount of invert sugar -

generally, in the preliminary work, 1.1875 grams - dissolving it in 75 cc. of distilled water in a 100 cc. Erlenmeyer flask. To this solution 5 cc. of concentrated hydrochloric acid were slowly added with stirring, and the flask set upon the steam bath and brought to a temperature of 67-69 degrees Cent. within 2½ to 3 minutes, and a temperature of 69 degrees maintained for 7 to 7½ minutes, making the total time of heating ten minutes. The solution was then rapidly cooled and neutralized with sodium hydroxide or sodium carbonate solution and diluted to the proper volume (500 cc.) after the addition of a few cc. of a saturated aqueous solution of thymol to prevent fermentation. (Thymol was found to have no reducing action upon Fehling's Solution).

Fifty cc. of the above solution were placed by means of a calibrated pipette into a 400 cc. Non-Sol beaker and 50 cc. of the Fehling's Solution, previously mixed and filtered, were added. This mixture was then covered with a watch glass and placed over a Bunsen flame on an asbestos gauze and flame so regulated that boiling began in 4 minutes and continued for exactly 2 minutes. The beaker was then removed and the cuprous oxide precipitated was filtered into a Gooch crucible, using suction. The crucible was prepared as described by Munson and Walker but washed with water at 60 degrees using strong suction; then washed with acetone and dried to constant weight.

Acetone was used instead of alcohol and ether as prescribed by Munson and Walker because it is less volatile and does not "blow up" the pad like ether when the latter comes into contact with a warm container. Many unpublished tests with routine work carried out in the Philippine College of Agriculture have shown

that results obtained by washing the cuprous oxide precipitate with acetone, are identical with those obtained by washing with alcohol and ether. To make the method more uniform the precipitate was always washed with 100 cc. of distilled water at about 60 degrees and with 30 cc. of pure acetone. The crucible was then placed in an electric oven heated to 100 degrees Cent. and dried to constant weight.

In an effort to obtain the most satisfactory length of time for drying the precipitates, a series of blank determinations were run from which changes of weight of the precipitates were plotted against the time of drying, on cross section paper. By observing the curves (Fig.1) it will be seen that after one hour's drying, the change is the slightest i.e. the results are practically constant between 1 and 1½ hour's drying so that weights obtained after one hour's drying may be considered as constant.

These curves also serve another purpose, that of blank determinations.

Munson and Walker used the method which is quoted from them as follows:

"In this work each one of us ran two blanks each day that determinations were made, and the results of each worker are corrected by his blanks for that day. In this way proper correction is made for the cuprous oxide precipitated spontaneously as well as for any loss of asbestos through solvent action of the alkaline liquid."[4]

This method of determining blanks is obviously far from ideal, because two blanks do not determine the correction for a whole set of determinations. A more proper method would be to run

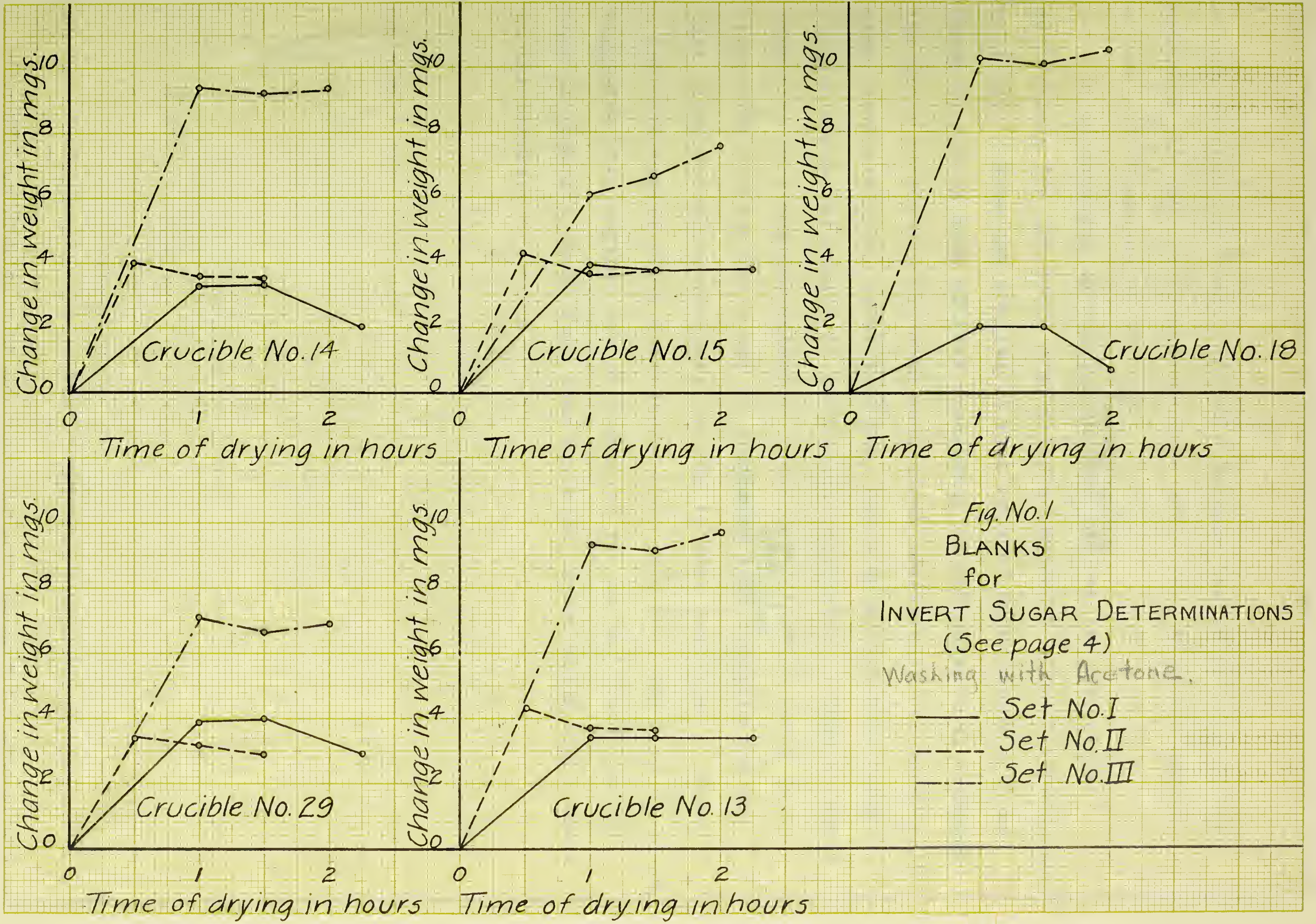

Fig. No. 1
BLANKS
for
INVERT SUGAR DETERMINATIONS
(See page 4)
Washing with Acetone.

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blanks on each individual crucible because the solubility of the asbestos pad and its consequent loss would vary with the amount of use and thickness of the pad.

In an endeavor to ~~accomplish this idea of~~ determine the individual corrections for the Gooch crucibles, a series of blank determinations were run. From the results of four sets of such determinations (page 8) run on each of five crucibles, it was shown that although the blank correction for each individual crucible is not exactly the same, each crucible, never-the-less, has a quite definite range through which the amount of correction oscillates. Thus it would seem that more logical method of blank determination would be to obtain the average for a set of blanks on *each crucible* and use that blank for all determinations run in that crucible. After a definite number of determinations have been run, say 5 or 6, it is suggested that another set of blanks be run on the crucibles and thus an accurate check kept upon any wide change which might occur in the blanks.

Below are recorded a few results showing differences due to various methods of proceedure and a comparison given with the results found in Munson and Walker's table.

The Cold Method of Inversion

Jan. 9, 1917.

Cu_2O Ppt.		
		2.3750 grams of sucrose
.2598 gram	Results of M. & W.	inverted, 25 cc. portions
.2536 ..	.267	used - equivalent to
		125 mgs. of invert sugar.

. . . .

Jan. 16, 1917.

Cu_2O Ppt.		
.2662 gram		
.2733 ..		2.3783 grams of sucrose
.2594 ..		
.2601 ..		treated exactly as
.2652 ..		
.2626 ..	Results of M. & W.	described above.
.2682 ..		
.2600 ..		
Av. .2619 ..	.267 gram	

. . . .

Jan. 23, 1917.

Cu_2O Ppt.		
		Treated as
.2639 gram	Results of M. & W.	
.2650 ..		above
Av. .2645 ..	.267 gram	

The Hot Method of Inversion

Jan. 24, 1917.

Cu$_2$O Ppt.	(Corrected by a common blank- 1.3 mg. added)	Result of M. & W.
.2635 gram	.2648 gram	
.2611 ..	.2624 ..	
.2625 ..	.2638 ..	
.2640 ..	.2653 ..	
.2635 ..	.2648 ..	
Av. .2629 ..	Av. .2642 ..	.267 gram

1.1875 grams of sucrose inverted and 50cc. portions used.

……

Cu$_2$O Ppt.
.2625 gram
.2664 ..
Av. .2644

1.1875 grams of sucrose inverted 50 cc. portions, equivalent to 125 mgs. invert sugar, used.

Same solution of sugar as above but alkaline content of Fehling's solution raised to 107%.

Cu$_2$O Ppt.	(Corrected for pipette volume)	Results of M. & W.
.2653 gram	.2667 gram	
.2660 ..	.2673 ..	
.2668 ..	.2681 ..	
Av. .2661 ..	Av. .2673 ..	.267 gram

……

In the following determinations the same concentration of alkaline tartrate solution as used in the preceeding was here used. Note that, barring corrections, the results are materially increased by an increase in the alkalinity content. Hence a test on the ~~alkalinity~~ true NaOH content of the caustic soda used should be made.

Cu$_2$O Ppt.	(Correction for pipette and for a general blank- 1.3 mgs. added)
.2659 gram	.2687 gram
.2677 ..	.2705 ..
.2679 ..	.2707 ..
.2664 ..	.2682 ..
.2670 ..	.2698 ..
Av. .2669 ..	Av. .2695 ..

……

Cu$_2$O Ppt. (Treated exactly as above)

.2656 gram
.2681 ..
.2659 ..
.2678 ..
.2702 ..
.2681 ..
Av. .2674 ..

The correction for pipette and blank added to the average of the column to the left gives .2700 grams

……

Herewith are given the results of blank determinations run as described on page 5 of this paper.

SET I

Crucible No.	14	15	18	29	13
Grams change in weight after drying					
1 hour	.0033	.0039	.0020	.0039	.0034
1½ hours	.0033	.0037	.0020	.0040	.0034
2¼ hours	.0020	.0038	.0007	.0029	.0034

SET II

½ hour	.0040	.0043	not run	.0034	.0043
1 hour	.0036	.0037		.0032	.0036
1½ hours	.0035	.0037		.0029	.0036

SET III*

Crucible No.	14	15	18	29	13
Grams change in weight after drying					
1 hour	.0094	.0061	.0103	.0071	.0093
1½ hours	.0092	.0067	.0101	.0067	.0091
2 hours	.0094	.0076	.0106	.0069	.0097

SET IV

1 hour	.0017	.0006	(.0030) (loss)	.0000	not run
1½ hours	.0018	.0006		.0001	

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* The results here are high probably because the Fehling's Solution used had been standing mixed for three days previous to using. The point to be brought out that each individual crucible should have a different correction blank is none the less ~~important as is~~ indicated by the results of this set.

In order to test the use of blanks, using the method described on pages 4 and 5, and to test for other possible errors, five determinations were run on the reduction of Fehling's Solution by invert sugar in the presence of sucrose. For this purpose 1.1875 grams of sucrose were inverted by the hot method and 2.75 grams of sucrose added to the neutral solution of invert sugar making a total of .400 grams of total sugar in each 50 cc. portion taken from the 500 cc. stock solution. This sugar solution was made up on the afternoon of March 27, 1917 and the determinations run the same evening. The Cuprous-oxide precipitates were washed with acetone, dried one hour, and a pipette correction equivalent to 1.3 mgs. of cuprous oxide was added to each. In correcting for blanks, the averages of the three most reliable determinations from the sets recorded on pages 8 and 9 were used, applying the idea of individual correction to each crucible. The results after all corrections were applied are herewith given:

Crucible No.	Cu_2O Ppt.
29	0.2712 gram
15	0.2713 ..
13	0.2681 ..
14	0.2691 ..
18	0.2715 ..
Average	0.2702 ..

From the results obtained from Munson and Walker's table, by interpolation, the corresponding value for the amount of sugar used in the above, gives a value in cuprous oxide precipitate of .2698 gram, indicating a difference of only 0.0004 gram between

the two results.

III
Conclusion

It is to be understood that this paper is the result of only a portion of a work of which the writer intended to carry out but which was interrupted by his departure from the university at an early date. The work shows, however, that after eliminating one source of error after another from the proceedure, results were obtained which check very closely with those tabulated by Munson and Walker. Thus persons working independently by the Munson and Walker method can obtain close checks, if the precautions ~~which are~~ discussed in this paper~~,~~ are observed.

References

[1] Jour. Am. Chem. Soc. 28, 668 (1906)

[2] Ibid., ~~28~~ p 671

[3] Ibid., ~~28~~ p. 451

[4] Ibid., ~~28~~ p. 667

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